Applied Surface Science 258 (2012) 2324–2331

Contents lists available at SciVerse ScienceDirect

Applied Surface Science

journal homepage: www.elsevier.com/locate/apsusc

Study of temperature effects on the conduction and trapping of charges in the alkali-silicate glass under electron beam irradiation

S. Fakhfakh[a,**], B. Elsafi[a,*], Z. Fakhfakh[a], O. Jbara[b]

[a] LaMaCop, Faculté des sciences de SFAX, Route Soukra Km 3, BP 1171, C.P. 3000 Sfax, Tunisia
[b] GRESPI/Matériaux Fonctionnels, UFR Sciences, BP 1039, 51687 Reims Cedex 2, France

ARTICLE INFO

Article history:
Received 24 April 2011
Received in revised form
10 September 2011
Accepted 3 October 2011
Available online 10 October 2011

PACS:
61.80. Fe
61.82 Ms
68.49 Jk

Keywords:
SEM
Glasses
Charge effect
Temperature

ABSTRACT

A scanning electron microscope (SEM) is employed to investigate the temperature effect on the charging behaviour of alkali-silicate glasses under electron beam irradiation using electrostatic influence method (EIM). A modified special arrangement adapted to the SEM allows to study charging mechanisms and charge transport characteristics of these glasses using the simultaneous measurement of displacement and leakage currents. The trapping process during continuous electron irradiation can be directly determined by the EIM. The experimental results reveal that the charging ability of glasses decreases with increasing temperature. The variation of charge process has been confirmed by measuring the surface potential in response to the sample temperature. In this report, we introduce also the secondary electron emission (SEE) yield. It was found the strong dependence of the SEE yield on the temperature variation. The higher is the temperature and the lower is the SEE yield. The trapping ability is analyzed taking into account the regulation mechanisms involved under electron irradiation.

1. Introduction

Due to the rapid growth of technology a great variety of material is used nowadays in the electronics industry. We will confine ourselves, however, to one material in particular, i.e. silica glass. Silica glass is widely used in the electronic industry, the best known applications being in lamps and cathode-ray tubes. But silica glass is also used, e.g, in the manufacture of videorecorder heads and in the fabrication in optical waveguides for telecommunication. Most people think of glass as being a transparent, insulating and stable material. Unfortunately, glass is not always as stable as one hopes it to be. Due to the bombardment by electrons the screen of cathode ray tubes may discolour and evolve oxygen. Glass discoloration is also observed in fluorescent lamps. At higher temperatures as used in the manufacture of glass containing articles, a glass transform into an aggressive viscous liquid, which will interact with most other materials. In order to study all the abovementioned processes recourse has to be made to surface by samples analysis techniques. The analysis of insulating samples and glass in particular, however, imposes severe restraints on the procedure to be followed. Electron bombardment, as used in most surface analysis techniques, will give rise to charge build up at or near the sample surface. This often results in the field-induced migration of the mobile alkali ions in the glass. The charging may also alter the energies of the emitted secondary electrons, which may carry the analytical information, resulting in reduced and unstable signals. Consequently, the surface analysis of glasses will only be successful when it is possible to reduce the surface potential to a low and stable value. The strategy to be followed will depend on the specific technique chosen and the stress applied (temperature, electrical field, etc.) to the sample. This work is focused on the study of temperature effects on the behaviour of alkali-silicate glasses under order electron beam irradiation of kiloelectron volt by means of an arrangement based on electrostatic influence phenomenon [1–5]. Recently, much effort has been made to understand the charging mechanism and it is generally believed that the charge processes are related to the localized energy states within the band gap of an insulator, which arise from the defects such as impurities and dangling bonds [6,7] in the structure of the insulator. Therefore, it is interesting and important to determine the buildup charge and its distribution in the insulator and to understand the charge regulation mechanisms associated. For this, a modified

* Corresponding author. Tel.: +216 74 676 610; fax: +216 74 676 610.
** Corresponding author.
E-mail addresses: slimfakhfakh@yahoo.fr (S. Fakhfakh), bassem.elsafi@yahoo.fr (B. Elsafi).

0169-4332/$ – see front matter © 2011 Elsevier B.V. All rights reserved.
doi:10.1016/j.apsusc.2011.10.007

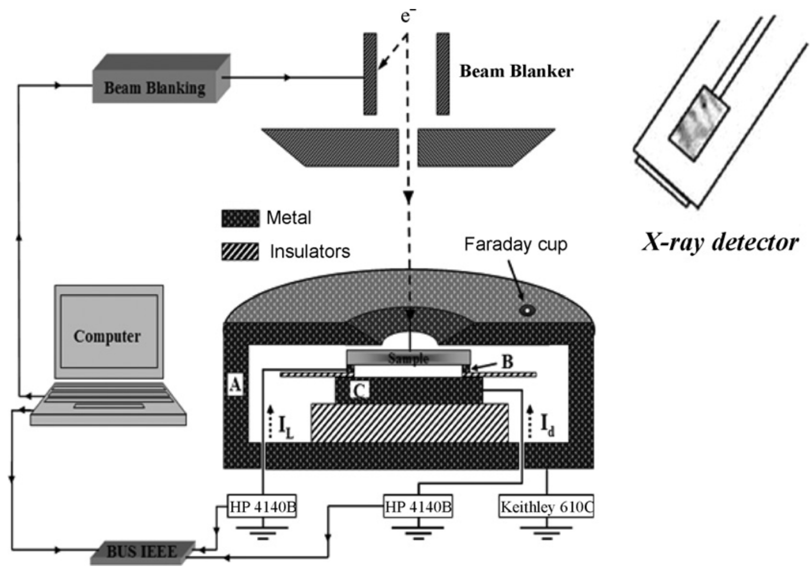

Fig. 1. Cut-away view of the experimental arrangement for measuring displacement and leakage currents.

special arrangement adapted to the SEM was used, that allows to measure not only the displacement current induced by trapped charge during and after electron irradiation, but also the leakage current resulting from the charges flow to the ground [4]. We have found that the charge carrier's mobility is highly dependent of the temperature. This behaviour is clearly verified with charging measurement results where increasing temperature enhances the charge carrier's mobility and decreases the amount of accumulated charges in the sample. The experimental results obtained are used to accurately determine the time constants of charging and discharging phenomena at each used temperature. In addition, the surface potential measurements deduced from X-ray spectra monitored with a X-ray spectrometer attached to the SEM confirm the charge state as a function of sample temperature. The secondary electron emission (SEE) yield of alkali-silicate glasses during electron irradiation is also determined by correlating the measured leakage and displacement currents. The results show that an increase of the temperature lead to an overall decrease of the magnitude of secondary electron emission yield at saturation.

2. Experimental and methods

2.1. Experimental device

The experiments were carried out in a SEM Philips 505 using a specific sample holder adapted to the SEM which is able to simultaneously measure both the displacement and the leakage currents (see Fig. 1). This arrangement was already described, in detail, in former publications [4] but here a modification was made to study the effect of the temperature. We thus will recall, in a synthetic way, its principle of operation and briefly described its geometry. (A) is a cylindrical grounded metallic enclosure with a circular hole of diameter 7 mm in its upper surface. The enclosure acts like a shield to prevent stray electrons (i.e. (SEs) and (BSEs) emitted from the SEM chamber-walls) to be collected by the probe disks and therefore to disturb the current measurements (displacement and the leakage currents). (C) Copper disk acting as an image charge probe connected to a highly sensitive picoammeter (HP4140B) for measurement of the displacement current, I_d. This copper disk (C) is set at the bottom of the enclosure on an insulating disk made in Teflon to avoid any electrical contact between the probe and the enclosure (A). The electrode intended for measuring the leakage current is a 0.5 mm thick frame-shaped electrode (B) made also in copper with a lateral dimension of (1 cm × 1 cm) and (0.9 cm × 0.9 cm) square opening. The electrode (B) is placed above the disk (C), on a 0.3 mm thick Teflon foil having the same square opening as (B) electrode. The Teflon foil guarantees the lack of electric contact between (B) and (C) plates. The metallic plate (B) is also connected to another picoammeter (HP4140B) to measure the leakage current I_L. Both picoammeters are interfaced to a PC. The HP4140B picoammeter is able to measure currents ranging from 10^{-15} A up to 1 mA with a basic accuracy of 0.5%. The current is directly monitored in integration mode (0.2 s).

The sample of parallelepiped shape (1 cm × 1 cm × 1 mm) placed under the opening of the enclosure (A), without touching it, is set on plate (B) with an intimate electric contact ensured by coating the periphery of the back surface of the sample with a 15 nm thick gold film. The bearing area (plate B) is a frame-shaped of 0.5 mm wide. The uncoated backside of the sample is separated from electrode (C) by a narrow vacuum gap of 0.8 mm.

It is worth emphasizing that this experimental arrangement differs from that operated for electrets [8] and others [2,9] in two points. In one hand, the rear electrode (C) is not in contact with the insulating layer so only the electrostatic influence current I_d is measured, whereas in other arrangements where insulator is in contact with the rear electrode, the radiation-induced conductivity (RIC) current, I_{RIC}, for instance, is superimposed to I_d. In the other hand, a second electrode in contact with the insulating layer is added to measure the so-called leakage current that may be due to the bulk and/or surface conduction processes, among others.

Thanks to a controlled heating system, connected to the electrode (B), the sample is brought to the desired temperature. The sample can be heated up to several hundreds of degrees during the experiment and the temperature is controlled by thermocouple on the metallic sample holder (electrode (B)), and a thermocouple on the surface of the glass (see Fig. 2). The specimen chamber of the SEM being under vacuum during the heating procedure.

In order to acquire X-ray spectra the SEM is equipped with a SiLi energy dispersive spectrometer (EDS). All the measurements were computer controlled and the measured data stored on a personal computer.

2.2. Samples and handling

The alkali-silicate glass samples provided from Saint-Gobain Company are used in this study. The samples are in the form of